

Abstract No. over651

## Structural Investigation of a Mixed-Valence Fe(II,III) 2D Layer Complex Below the Magnetic Ordering Temperature.

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Beamline(s): X3A1

**Introduction:** In the synthesis of trinuclear carboxylates - systems which have had our attention for a long time [1,2] - a very intriguing intermediate can be produced with formate as ligating group. The crystals of this compound differ significantly, both in color and in hardness, from those of the mixed valence trinuclear iron systems. From results of variable-temperature magnetic susceptibility measurements of this complex it has been established that a magnetic ordering takes place close to 20 K, and the present study is intended to examine the structural and electronic reasons for this effect.

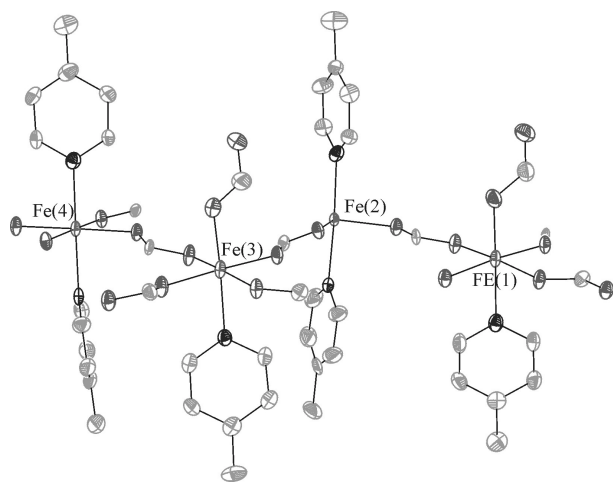
**Methods and Materials:** Synchrotron X-ray data were collected at beamline X3A1 at a crystal temperature of 16(5) K. 33398 integrated reflections reduced to 13696 unique reflections with  $R_{\text{int}}=0.026$ . The structure solution was impeded by the presence of pseudosymmetry, which indicated a centrosymmetric structure. However, the correct space group was confirmed to be P1.

**Results:** The compound,  $[\text{Fe}_2^{\text{II}}\text{Fe}_2^{\text{III}}(\text{HCOO})_{10}(\text{C}_6\text{H}_7\text{N})_6]$ , consists of formate-bridged  $\text{Fe}^{\text{II}}$  and  $\text{Fe}^{\text{III}}$  atoms alternately in an infinite 2D array, Figure 1. In the present compound, the  $\text{Fe}^{\text{III}}$  atoms have their octahedral coordination sphere completed by one formate and one  $\gamma$ -picoline in the axial positions, whereas the divalent irons are axially coordinated to two  $\gamma$ -picoline molecules. The interlayer distance is approximately 10 Å and neighbouring layers are only held together with weak  $\pi$ - $\pi$  interactions between axially coordinated  $\gamma$ -picoline ligands (Figure 2). The structural features at 16 K were found to closely resemble the structure at 120 K, which represents the structure above the magnetic ordering temperature. Currently we are pursuing the determination of the electron density distribution for this complex from the low temperature data.

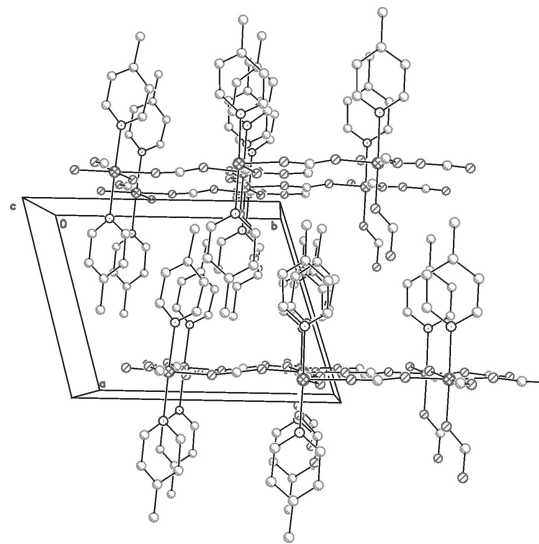
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### References:

- [1] C. Wilson, *et al.*, *J. Am. Chem. Soc.* (2000), 122, 11370-11379.
- [2] J. Overgaard, Ph. D. thesis, Department of Chemistry, University of Aarhus, Denmark, 2001.



**Figure 1.** Thermal ellipsoids plot of the title compound. Ellipsoids truncated at 50% probability level.



**Figure 2.** Packing plot showing the interlayer distance.